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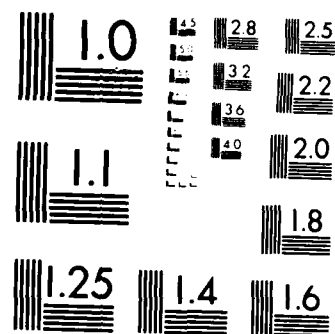
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TECHNICAL REPORT ARLCB-TR-85002

SOLID METAL INDUCED EMBRITTLEMENT OF METALS

M. H. KAMDAR

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US ARMY ARMAMENT RESEARCH AND DEVELOPMENT CENTER
LARGE CALIBER WEAPON SYSTEMS LABORATORY
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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Many ductile metals in intimate contact with thin coatings of low melting solid metal which exhibit liquid metal embrittlement, also manifest severe embrittlement when tested at temperatures below the melting point of the coating. A significant decrease in stress, strain, and reduction-in-area occurs at fracture. Fracture propagates fast, but at a rate which is one or two orders of magnitude slower than that in liquid metal environments. Fracture occurs by

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20. ABSTRACT (CONT'D)

brittle intergranular or transcrystalline mode with multiple cracks and
branching. This new phenomena is known as solid metal induced embrittlement
of metals (SMIE). SMIE also occurs when the embrittling metal is present as
an internal environment in the base metal such as inclusions. This report
describes the occurrence of SMIE in metals and alloys used in industry and
presents results of recent investigations. It describes the effects of time,
temperature, and stress on SMIE. It discusses the occurrence, the mechanisms
of SMIE, and its similarity to LME. This new phenomena must be considered
while investigating environmentally induced failure of failure analysis of
metals and alloys.

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INTRODUCTION

In certain liquid metal embrittlements (LME) couples, embrittlement occurs below the melting temperature of the solid. The severity of embrittlement increases with increasing temperature with a sharp and significant increase in severity at the melting point (m.p.) of the embrittler (ref 1), Figure 1. Above the m.p., embrittlement has all the characteristics of liquid metal embrittlement. The occurrence of embrittlement below the m.p. of the embrittling species is known as solid metal induced embrittlement of metals, or SMIE.
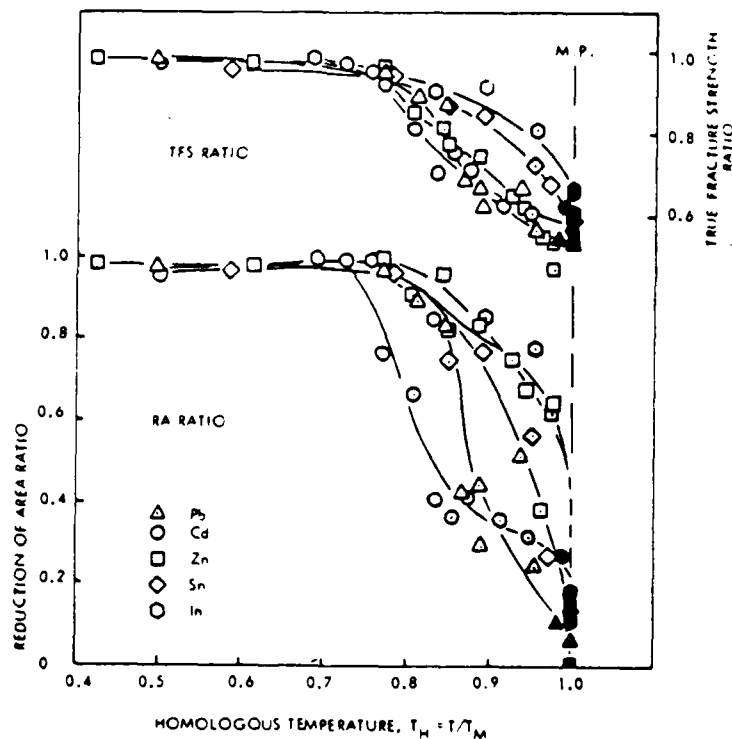

Figure 1. Comparison of normalized true fracture strength and reduction-in-area as a function of homologous temperature (T, T_M, T_N are absolute test temperature, melting temperature, and 0.75 of melting temperature, respectively (from Lynn et al (ref 1)).

References are listed at the end of this report.

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Although solid metal induced embrittlement of metals (SMIE), per se, has not been mentioned or recognized as the embrittlement phenomena in industrial processes, many reports of loss in ductility, strength, and brittle fracture of metals and alloys have been reported by electroplated metals and coatings or inclusions of low melting metals below their m.p. Kennedy (ref 2) observed delayed failure of cadmium plated high strength steel below the m.p. of cadmium. Accordingly, cadmium plated steel bolts, in spite of their excellent resistance to corrosion, are not recommended for use above 450°F. Hildebrand (ref 3) observed cracks in notched tensile specimens of various steel by solid cadmium. Iwata et al (ref 4), Fager and Spurr (refs 5,6), Meyn (ref 7), Stoltz and Stuben (ref 8) have reported solid cadmium embrittlement of steel, titanium, and also of titanium by silver and gold. Mostovoy and Breyer (ref 9) reported loss in ductility in leaded steels below the m.p. of lead and accounted for numerous elevated service failure of leaded steel such as radial cracking of gear teeth during induction hardening heat treatment, fracture of steel shafts during straightening at elevated temperature, and heat treatment failure of jet engine compressor discs. Grubb (refs 10,11) reported that both liquid and solid cadmium dissolved in inert non-embrittling coolant liquid cesium embrittles zircoloy-2 nuclear fuel cladding tubes used in nuclear metals. Collipriest (ref 12) reported cracking of Inconel vacuum seals by solid cadmium. All these reports of brittle failure clearly indicate the importance of SMIE in industrial processes.

The recognition and investigation of SMIE was made first in 1967 by Iwata et al (ref 4) who studied delayed failure of steels in solid cadmium environment. Subsequently, Fager and Spurr (refs 5,6) in 1970 reported

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embrittlement of steel and titanium by solid cadmium. They detected cadmium on fracture surfaces near the crack tip and suggested that vapor transport to the propagating tip is necessary for embrittlement. Meyn (ref 7) reported embrittlement of titanium by cadmium. In 1974 Warke, Gordon, Breyer, and their co-workers at the Illinois Institute of Technology made a systematic investigation of SMIE of steel by a number of solid metal embrittling species (see Figure 1), concluding that SMIE was a generalized phenomena of embrittlement at least in steel. Thus, solid metal as an external environment can cause embrittlement. In 1969, Mostovoy and Breyer (ref 9) were the first to recognize that SMIE can occur when the embrittling solid is an internal environment, i.e., present in the solid as an inclusion. They clearly demonstrated that internally leaded steel is embrittled below the m.p. of lead inclusion in steel. Iwata et al (ref 4) made the preliminary investigations of the mechanism of the SMIE and suggested that formation of intermetallic compounds was responsible for SMIE. Fager and Spurr (refs 5,6) suggested vapor transport of embrittler to crack tip as the mechanism for the cracking process. Mostovoy and Breyer (ref 9) suggested that "reduction in cohesion" mechanism proposed for LME (refs 13,14) can also account for SMIE, but the kinetics of the cracking processes are determined by the thermally activated processes such as the sticking of the embrittling species at the crack tip. The most recent and substantial work on the mechanisms of SMIE is that by Gordon (ref 15) and Gordon and An (ref 16) who suggest stress induced diffusion penetration by the embrittling species in the grain boundary causes crack initiation, whereas multi-layer self-diffusion of embrittler controls crack propagation.

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The brittle fracture in LME and SMIE is of significant scientific interest because the embrittling species are in the vicinity or at the tip of the crack and are not transported by dislocations or by slip due to plastic deformation into the solid as is hydrogen in hydrogen embrittlement of steels. Also, embrittling species are less likely to be influenced by the effects of impurities in the grain boundaries such as antimony, phosphorous, tin, etc., which cause significant effects on the severity of hydrogen and temper embrittlement of metals. For these reasons, investigations of SMIE and LME are more unambiguously interpretable than similar effects in other environments such as hydrogen and temper embrittlement of metals. Thus, solid-liquid environmental effects provide a unique opportunity to study embrittlement mechanisms in a simple and direct manner under controlled conditions. It is believed that a common mechanism may underlie solid, liquid, and gas phase induced embrittlement. The interactions at the solid environment interface and the transport of the embrittling species to the crack tip may characterize a specific embrittlement phenomena. A study of SMIE and LME may provide insights into the mechanisms of hydrogen and temper embrittlement. It is apparent that the phenomena of SMIE is both of industrial and scientific importance. In this report we will review the investigations on the occurrence and the mechanisms of SMIE.

CHARACTERISTICS OF SMIE

So far SMIE has been observed only in those couples in which LME occurs suggesting that LME is a prerequisite for the occurrence of SMIE. SMIE may occur in the absence of LME if a brittle crack cannot be initiated at the

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m.p. of the embrittler. A recent compilation of SMIE couples is given in Tables I and II (ref 17) where it is seen that all solid metal embrittlers are known to cause liquid metal embrittlement. SMIE and LME are a strikingly similar phenomena. The prerequisites for SMIE are the same as those for LME. Thus, the requirements are: (1) intimate contact between the solid and the embrittler, (2) presence of tensile stress, (3) crack nucleates at the solid embrittler interface from a barrier such as a grain boundary, and (4) the presence of embrittling species at the propagating crack tip. Also, metallurgical factors that increase brittleness in metals such as grain size, strain rate, increase in yield strength, solute strengthening, presence of notch or stress raiser, etc., all appear to increase embrittlement. The susceptibility to SMIE is stress and temperature sensitive and does not occur below a specific threshold value. Embrittlement by delayed failure is also observed for both LME and SMIE (Table III).

However, some differences also exist. Multiple cracks are formed in SMIE whereas in LME usually a single crack propagates to failure. The fracture in SMIE is propagation controlled. However, crack propagation rates are at least two to three orders of magnitude slower than in LME. Brittle intergranular fracture changes to ductile shear mode because of the inability of the embrittler to keep up with the propagating crack tip. Incubation periods have been reported indicating that the crack nucleation process may not be the same as in LME. Nucleation and propagation are two separate stages of fracture in SMIE. These differences may arise because of the rate of reaction or interactions at metal-embrittler interface but most importantly, because the transport properties of solid and liquid metal embrittler are of significantly

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TABLE I. OCCURRENCE OF SMIE IN STEELS
(from Druschitz and Gordon (ref 17))

Base Metal	Embrittler (melting point)	Onset of Embrittlement	Test Type	Specimen Type
1041	Pb (327°C)	288°C	ST	S
1041 leaded	Pb	204°C	ST	S
1095	In (156°C)	100°C	ST	S
3340	Sn (232°C)	204°C	ST	N
	Pb	316°C	ST	N
4130	Cd (321°C)	300°C	DF	N
4140	Cd	300°C	DF	N
	Pb	204°C	ST	S
	Pb-Bi (NA)	below solidus	ST	S
	Pb-Zn (NA)	below solidus	ST	S
	Zn (419°C)	254°C	DF	N
	Sn	218°C	DF	N
	Cd	188°C	DF	N
	Pb	160°C	DF	N
	In	room temp	DF	N
	Pb-Sn-Bi (NA)	below solidus	ST	S
	In	80°C	DF	S
	Sn	204°C	ST	S
	Sn-Bi (NA)	below solidus	ST	S
	Sn-Sb (NA)	below solidus	ST	S
	In	110°C	DF	S
	In	93°C	DF	S
	In-Sn (118°C)	93°C	DF	S
4145	Sn	204°C	ST	S
	In	121°C	ST	S
	Pb-4wt%Sn (NA)	204°C	ST	S
	Pb-Sn (NA)	204°C	ST	S
	Pb-Sb (NA)	204°C	ST	S
	Pb	288°C	ST	S
4145 leaded	Pb	204°C	ST	S
4340	Cd	260°C	DF	N
	Cd	300°C	DF	N
	Cd	38°C	DF	S
	Zn	400°C	DF	N
4340M	Cd	38°C	DF	S
8620	Pb	288°C	ST	S
8620 leaded	Pb	204°C	ST	S
A-4	Pb	288°C	ST	S
A-4 leaded	Pb	204°C	ST	S
D6ac	Cd	149°C	DF	N

DF - Delayed Failure Tensile Test   ST - Standard Tensile Test
N - Notched Specimen   S - Smooth Specimen
NA - No Available Data

different magnitudes. It has been suggested that reductions in the strength of cohesive strength of atomic bonds at the tip is responsible for both SMIE and LME (refs 13,14). However, transport of embrittler is definitely the rate controlling factor in SMIE. Another possibility is that stress assisted penetration of embrittler in the grain boundaries initiates crack, whereas surface self-diffusion of the embrittling species similar to that proposed for LME controls crack propagation (refs 15,16). We will discuss these mechanisms later.

TABLE II. OCCURRENCE OF SMIE IN NON-FERROUS ALLOYS
(from Druschitz and Gordon (ref 17))

Base Metal	Embrittler (melting point)	Onset of Embrittlement	Test Type	Specimen Type
Ti-6Al-4V	Cd (321°C)	38°C	DF	S
	Cd	149°C	BE	S
Ti-8Al-1Mo-1V	Cd	38°C	DF	S
	Cd	149°C	BE	S
Ti-3Al-14V-11Cr	Cd	149°C	BE	S
Ti-6Al-6V-2Sn	Cd	149°C	BE	S
	Ag (961°C)	204 to 232°C	BE	S
	Au (1053°C)	204 to 232°C	BE	S
Cu-Bi (UNSEG)	Hg (-39°C)	-84°C	ST	S
Cu-Bi (SEG)	Hg	-87°C	ST	S
Cu-3%Sn (SEG)	Hg	-48°C	ST	S
Cu-1%Zn (SEG)	Hg	-46°C	ST	S
Tin-Bronze	Pb (327°C)	200°C	IM	S
Zinc	Hg	-51°C	ST	S
Inconel	In (156°C)	room temp	RE	S
Zircoloy 2	Cd	300°C	ST	S

SEG   — Heat Treated to Segregate Solutes to Grain Boundaries
UNSEG — Heat Treated to Distribute Solutes Uniformly
DF   —   Delayed Failure Tensile Test
IM   —   Impact Tensile Test
RE   —   Residual Stress Test
S   —   Smooth Specimen
ST   —   Standard Tensile Test
BE   —   Bend Test
N   —   Notched Specimen

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TABLE III. DELAYED FAILURE IN SMIE AND LME SYSTEMS
(from Gordon (ref 15))

Type A Behavior - Delayed Failure Observed		
Base Metal	Liquid	Solid
4140 steel	Li	Cd
4340 steel	Cd	In
4140 steel	In	Cd
4140 steel		Pb
4140 steel		Sn
4140 steel		In
4140 steel		Zn
2024 Al	Hg	
2424 Al	Hg-3 pct Zn	
7075 Al	Hg-3 pct Zn	
5083 Al	Hg-3 pct Zn	
Al-4 pct Cu	Hg-3 pct Zn	
Cu-2 pct Be	Hg	
Cu-2 pct Be	Hg	
Type B Behavior - Delayed Failure Not Observed		
Base Metal	Liquid	Solid
Zn	Hg	
Cd	Hg	
Cd	Hg + In	
Ag	Hg + In	
Al	Hg	

INVESTIGATIONS OF SMIE

First investigations of delayed failure were reported by Iwata et al (ref 4) in cadmium, zinc, and indium plated notched tensile specimens of 4340, 4130, 4140, and 18% Ni maraging steel in the 200 to 300°C temperature range (Figures 2 and 3). The results indicate that 4340 is most susceptible and 18% Ni maraging steel was the least susceptible alloy to cadmium embrittlement. They noted that crack propagation was discontinuous but crack nucleation was immediate, i.e., incubation did not exist for crack nucleation. Embrittlement was not observed in indium plated steels. The activation energy for steel-cadmium embrittlement was 39 Kcal/mole (Figure 4) and corresponded to
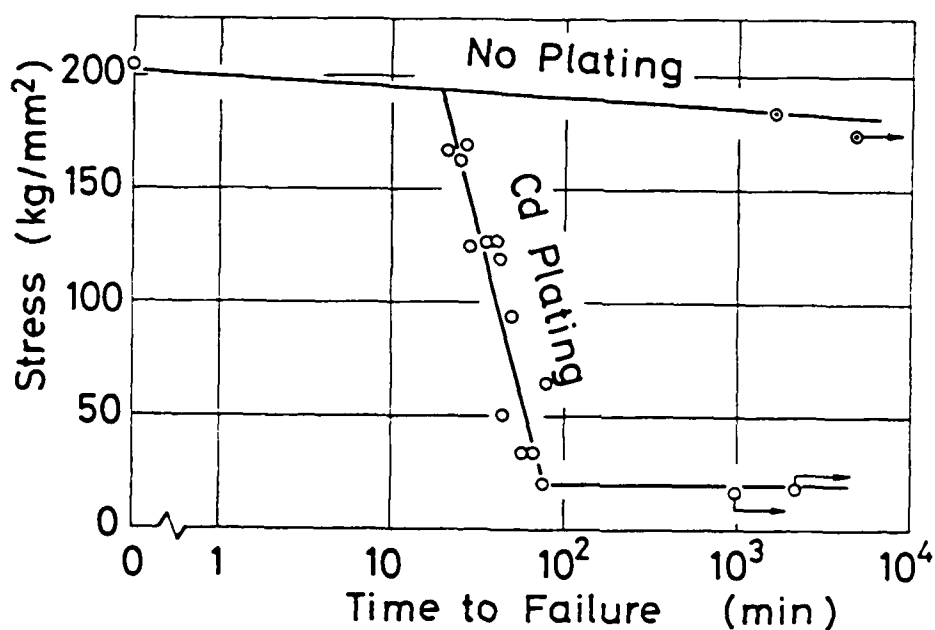
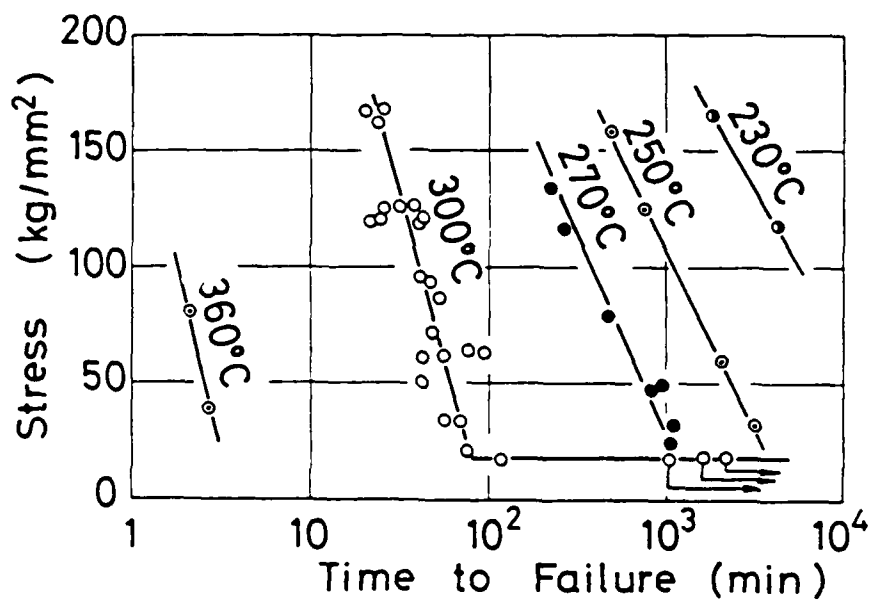Figure 2. Delayed failure of cadmium plated 4340 steel at 300°C (from Asayama (ref 20)).

Figure 3. Delayed failure of cadmium plated 4340 steel at various temperatures (from Asayama (ref 20)).
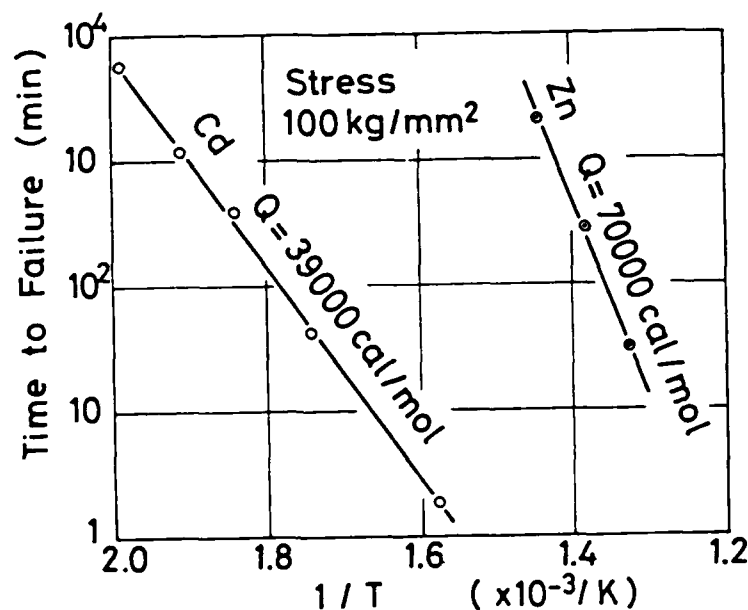
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Figure 4. Arrhenius plots of delayed failure for cadmium and zinc plated
4340 steel (from Asayama (ref 20)).

diffusion of cadmium in the grain boundary. They also reported that plating

of a thin layer of nickel or copper acts as barriers to embrittler and

prevents SMIE of steel. Contrary to the report of Iwata et al (ref 4), Gordon

and An (ref 16) reported that solid indium embrittles steels and an incubation

period exists for crack nucleation. Mostovoy and Breyer (ref 9) and Warke and

Breyer (ref 18) investigated embrittlement of different steels in lead both as

external and internal (leaded steel) environments and conclusively

demonstrated that SMIE is a reproducible effect. They also demonstrated that

SMIE is an extension of LME. Internally, leaded high strength steels are

susceptible to severe embrittlement in the range of the m.p. of lead and this

embrittlement is a manifestation of LME. However, it was found that onset of

embrittlement occurs some 200°F below the m.p. of lead (625°F) and is

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continuous up to the m.p. with no discontinuity or anomalies in the variation in the embrittlement with temperature. At the m.p. of lead, a sharp increase occurs in the severity of embrittlement and brittle ductile transition occurs in the temperature of 370°C to 450°C, Figure 5. The same behavior was noted for pure lead as an external environment soldered on to 4140 steel, Figure 1. Lynn et al (ref 1) have extended such embrittlement of surface coating of solid zinc, lead, cadmium, tin, and indium on steel. The embrittlement manifests itself as a reduction in tensile ductility over a range of temperature extending from about three-quarters of the absolute m.p. of the embrittler and up to the m.p., Figure 1. It was shown that embrittlement is caused by the growth of stable subcritical intergranular cracks and that crack propagation is the controlling factor in embrittlement. This indicated that transport of embrittlers to the crack tip was either by vapor phase or by surface or volume diffusion. The vapor pressures of the five embrittling species at the m.p. varied widely and ranged from 10^{-6} to 10^{-26} torr (Table IV). However, the crack propagation times for all the five embrittlers were similar. The estimated values of the diffusion coefficients ranged in the vicinity of 10^{-4} to 10^{-6} cm^2/sec (ref 15). These values are comparable to surface or self-diffusion of embrittler over embrittler, and suggest diffusion rather than vapor transport as the rate controlling process.
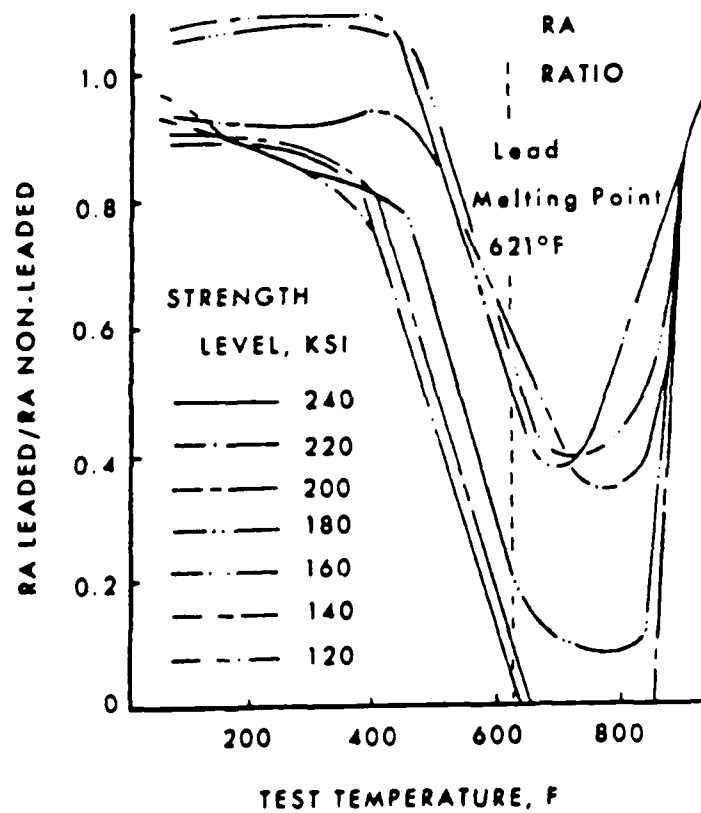
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Figure 5. Ratio of reduction-in-area for leaded to non-leaded steel at
various temperatures (from Mostovoy and Breyer (ref 9)).

TABLE IV. EMBRITTLER VAPOR PRESSURES AND CALCULATED VAPOR TRANSPORT TIMES
AT THE EMBRITTLER MELTING TEMPERATURES (from Gordon (ref 15))

Embrittler	Vapor Pressure, kPa	t,s	
Zn	2×10^{-2}	5×10^{-3}	
Cd	1×10^{-2}	1×10^{-2}	
Hg (at R.T.)	3×10^{-4}	3×10^{-1}	
			1 s
Sb	4×10^{-7}	3×10^{2}	
K	1×10^{-7}	1×10^{3}	
			1 h
Na	2×10^{-8}	5×10^{3}	
TI	4×10^{-9}	3×10^{4}	
			1 day
Pb	5×10^{-10}	2×10^{5}	
Bi	2×10^{-11}	5×10^{6}	
Li	2×10^{-11}	5×10^{6}	
			1 yr=3×10^{7}
In	3×10^{-22}	3×10^{17}	
			10^{10} yrs
Sn	8×10^{-24}	1×10^{19}	
Ga	6×10^{-39}	2×10^{35}	

(1 atm $\cong 10^{2}$kPa)

DELAYED FAILURE AND MECHANISM OF SMIE

If a metal in contact with the embrittling species is loaded to a stress which is lower than that to fracture and tested at various temperatures, then either the environment-induced fracture initiates and propagates instantaneously, e.g., Zn-Hg LME couple; or such a failure occurs after sometime, i.e., delayed failure or static fatigue is observed. Examples of these two types of fracture process are given in Table III (ref 15). Investigations of delayed failure provides an opportunity to separate crack nucleation from crack propagation, but specifically to evaluate transport related role of the embrittling species on embrittlement. SMIE is a propagation controlled fracture process and the time, temperature, and stress dependence of

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embrittlement presents an opportunity to study the kinetics of the cracking process.

The most widely investigated embrittlement system is 4140 steel by solid and liquid indium (refs 15,19,20). The detailed investigations in this system are reported by Gordon and An (ref 20). They used an electrical potential drop technique to monitor crack initiation and propagation and investigated the effects of temperature and stress level on delayed failure in 4140 steel by liquid and solid indium. The effects of temperature and stress level on the initiation time (incubation period for crack initiation) for both SMIE and LME are given in Figure 6. The activation energy of crack initiation process is ~ 37 Kcals/mole, and is essentially independent of the applied stress level. The activation energy is considered to represent the energy for stress-aided self-diffusion of indium, in both liquid or solid state in the grain boundary of the steel. Thus, crack initiation in both SMIE and LME occurs first by the adsorption of the embrittler at the site of crack initiation. However, because of the presence of the incubation period, the rate controlling process is stress-aided diffusion penetration of the base metal grain boundaries and is a process similar to that proposed by Kristal (ref 21). Such penetration somehow reduces the stress necessary to initiate a crack causing embrittlement of the base metal (ref 16). This is an interesting mechanism. However, we still do not know how and why the embrittler diffused in the grain boundary reduces the stress or have experimental evidence for the presence of such penetration of the embrittler in the grain boundaries. Therefore, on this basis it will be difficult to explain embrittlement of single crystals which do not contain grain
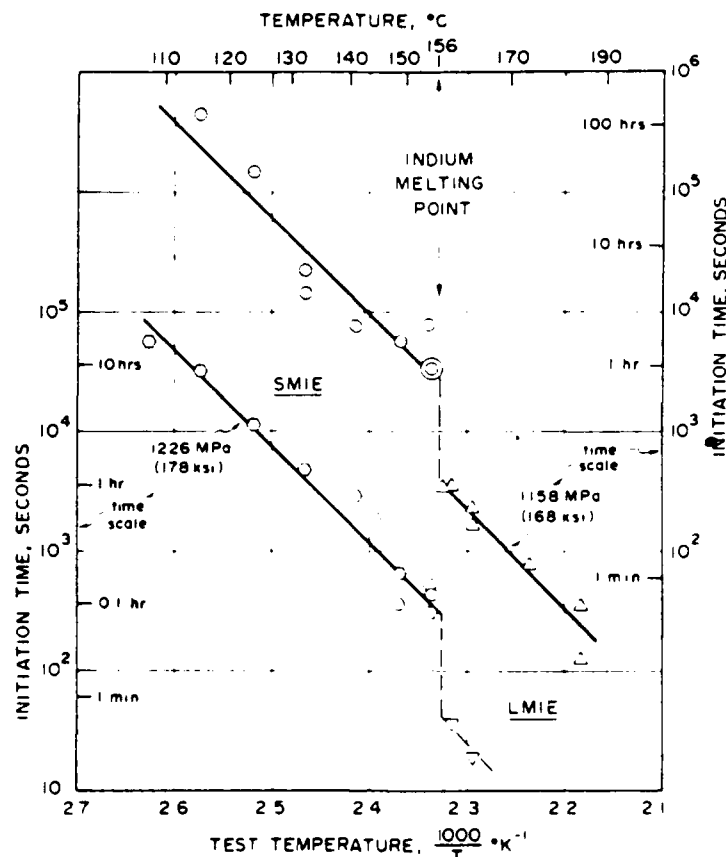
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Figure 6. Initiation time vs. temperature in SMIE and LME of 4140 steel in indium at two stress levels (from Gordon and An (ref 16)).

boundaries. It is still possible to suggest that embrittlement reduces

cohesion of atoms in the grain boundaries and thus is in accord with the

"reduction in cohesion" mechanism. The crack propagation time as a function

of temperature and stress level for both SMIE and LME of steel by indium are

plotted in Figure 7. The activation energy for crack propagation in SMIE was

5600 cals/mole and this represents the energy for self-diffusion of indium

over indium. Thus, propagation is controlled by the diffusion of indium over

several multi-layers of indium adsorbed on the crack surface or by the

so-called waterfall mechanism of embrittlement (ref 1). This mechanism is a

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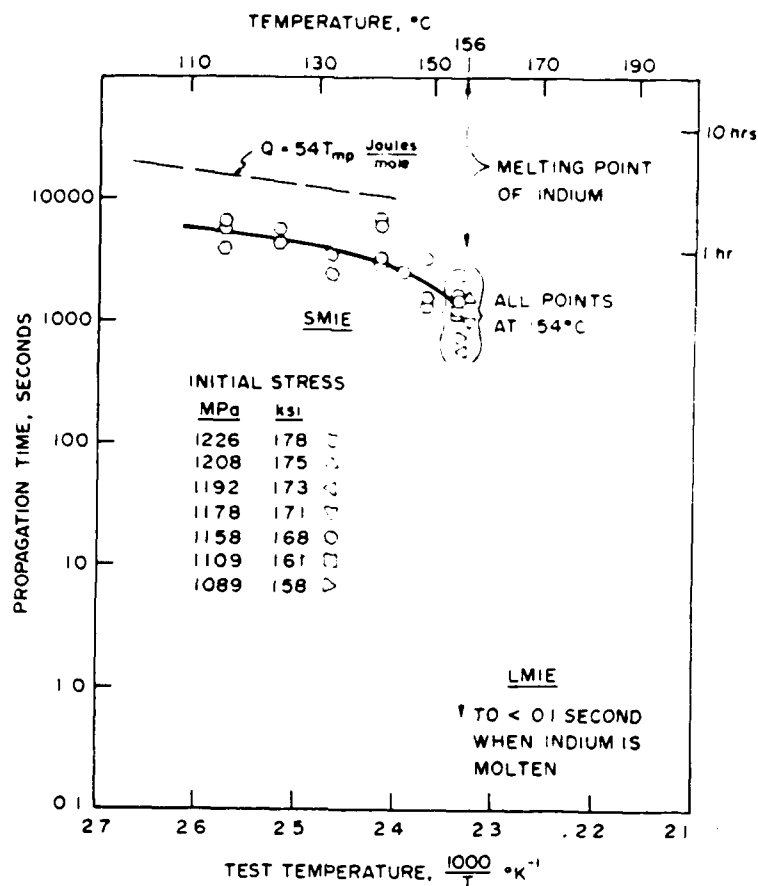
Figure 7. Propagation time vs. temperature in SMIE and LME for 4140 steel in indium environment at various initial stress levels.

variation of that proposed and discussed in detail as the "reduction in cohesion" mechanism for LME by Westwood and Kamdar (ref 13) and Stoloff and Johnston (ref 14). In summary, adsorption of the embrittler at the solid surface is followed by the rate controlling step of stress-aided diffusion of the embrittler in the grain boundary of the base metal. Somehow, the embrittler reduces cohesion of the grain boundary base metal atoms thereby initiating a crack at a lower stress with the result that embrittlement occurs. Crack propagation occurs by multi-layer self-diffusion of the

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embrittling species. These processes are valid for both LME and SMIE in the steel-indium embrittlement couple. It is clear that SMIE is similar to LME except that SMIE is an embrittler transport limited process. In this regard, study of SMIE is important in eliminating the possibility in LME that once nucleated a crack may propagate in a brittle manner in the absence of the embrittling species at the tip of the crack. SMIE is a new and recent phenomena and many more investigations are needed concerning the effects of metallurgical, mechanical, and chemical parameters on embrittlement.

SUMMARY

SMIE failures have been reported by engineers in industrial metals and alloys. Scientific investigations of this phenomena have clearly established it as yet another embrittlement process similar to LME. SMIE is somewhat less severe and fracture propagation rates are slower than that for LME. Besides this, both SMIE and LME are quite similar and appear to occur by similar mechanisms. Cracks may initiate by either "reduction in cohesion" mechanisms or by stress-induced diffusion of embrittler in the grain boundaries of the base metal. However, crack propagation in both SMIE and LME occurs by surface diffusion of the embrittler over adsorbed layers of the same species on the crack surfaces. More investigations similar to those reported for LME are needed. However, it is clear that metallurgists, engineers, etc., must recognize this as yet another phenomena of environmental induced embrittlement.

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